Through this treatment with enzyme, the hair becomes easily removable from the hide by just light rubbing. After removing the hair, the hide is washed three times in 100 *l.* of 5% salt solution and is thereafter further washed for 10 hours in running water. When the hide after having been thus subjected to temperature treatment, enzyme treatment, thereafter to removal of hair and to washing, is immersed in 120 *l.* of 0.5% acetic acid solution and agitated vigorously for 6 hours, all the collagen fibers are easily dispersed into a colloidal solution. At this time, the total amount of solution is about 150 *l.* and its collagen concentration is 0.8%. After this colloidal solution is separated centrifugally or filtrated by a filter press using cloth or pulp as a filter-aid and thereafter neutralized with ammonia to make pH 6–8, there occurs a complete reconstitution of fibers. The fibers thus reproduced are collected by means of centrifugal separation, washed with water and thereafter dried with acetone to obtain 12 kg. of pure white dry fibers.

The turbid colloidal solution of collagen obtained by the above-mentioned process may be changed into a clear monomolecularly dispersed solution by digesting the 150 *l.* of colloidal solution with 30 g. of pepsin at 25° C. for 24 hrs. This solution is then clarified centrifugally or filtrated and thereafter neutralized with ammonia to pH 6–8 to reconstitute fibers completely. The fibers thus reconstituted are then collected by centrifugal separation, washed with water and dried with aceton to obtain 12 kg. of pure white collagen fibers. The difference between the reconstitution of fibers directly from the turbid colloidal solution in which the molecules are dispersed in an aggregate form and that of fibers from the clear monomolecularly dispersed solution is that the former is hardly soluble in hot water (60°–70° C.), whereas the latter is easily soluble, so that the latter fibers are suitable for the manufacture of gelatin. While, in this example, the steer hide immediately after slaughtering is used as raw material, the same result can be obtained with cured hide, the limed unhaired hide and with osein prepared from animal bone. However, in treatment of limed hide, even after the neutralization of calcium hydroxide, the shrinkage temperature lowers to 55° C., so that unless the lowest shrinkage temperature is measured in advance according to the kind and state of raw material and the pretreatment is effected at that temperature, the loss of collagen fibers at the stage of enzyme treatment becomes large. While in this example, trypsin was used for enzyme treatment, the same result can be obtained by using any other proteolytic enzymes, other than collagenase, having the enzyme activity at pH 5–9, that is pancreating and, chymotrypsin. The same results are obtained if the clear monomolecularly dispersed solution from the turbid colloidal solution, is treated with a proteolytic enzyme other than pepsin having the activity at pH 4.5–1, for example, the enzymes extracted from fungi. When any acids are used for extraction, other than acetic acid, for example, inorganic acid such as hydrochloric acid, sulfuric acid, etc., or organic acid such as citric acid, malonic acid, lactic acid, etc., at pH 4.5–1.5, the same results are accomplished.

Example 2

This example refers to the use of protein denaturating agents at room temperature as pretreatment. In the same way as described in Example 1, 30 kg. of steer hide washed with a salt solution and running water is immersed in a 2.5 M potassium thiocyanate solution or a 3 M calcium chloride solution for 24 hours at 25° C. and thereafter washed in running water for 24 hours to remove the denaturing agents. Again, in quite the same way as described in Example 1, the enzyme treatment, washing and acid extraction are effected to obtain a turbid colloidal solution, followed by the neutralization, washing and drying to obtain 12 kg. of pure white reconstituted fibers. Also, in the same way as described in Example 1, the turbid colloidal solution is made into a completely clear monomolecularly dispersed solution by using pepsin, followed by the neutralization, washing and drying to obtain 12 kg. of reconstituted fibers. As the denaturing agents, bivalent inorganic salts such as barium chloride, magnesium chloride, etc. other than potassium thiocyanate and calcium chloride, sodium salicylate, guanidine hydrochloride, urea, etc. can be used to obtain the same result, but if the lowest concentration for the denaturation is not selected in respect of each thereof, the loss at the stage of enzyme treatment becomes large.

It should be understood, of course, that the foregoing disclosure relates to only preferred embodiments of the invention and that it is intended to cover all changes and modifications of the examples of the invention herein chosen for the purposes of the disclosure, which do not constitute departures from the spirit and scope of the invention as claimed.

What is claimed is:

1. A process for dispersing colloidally collagen fiber materials, which are normally insoluble in dilute acid, alkali and neutral salt solutions, into a fiber reproducible state, comprising the steps of; firstly, de-naturing the collagenous material by preheating it for 20 minutes to 63° C.; secondly, re-naturing the product of the first step by cooling it to 25° C. and immersing it in an acid buffer solution of pH 8; thirdly, treating the product of the second step with a proteolytic enzyme selected from a class consisting of trypsin, chymotrypsin, and pancreatin, for 48 hours, and fourthly, dispersing the product of the third step into 0.5% acetic acid solution for six hours for extraction.

2. A process for dispersing colloidally collagen fiber materials, which are normally insoluble in dilute acid, alkali and neutral salt solutions, into a fiber reproducible state, comprising the steps of: firstly, pre-de-naturing the collagenous material with a protein de-naturing agent selected from the group consisting of potassium thiocyanate and calcium chloride at 25° C. for 24 hours; secondly, re-naturing the product of step one by washing it in water until the de-naturing agents are removed; thirdly, treating the product of step two with a proteolytic enzyme selected from a class consisting of trypsin, chymotrypsin, and pancreatin for 48 hours; and fourthly dispersing the product of step three into 0.5% acetic acid solution for six hours for extraction.

3. A process for preparing a molecularly dispersed fiber reproducible collagen solution normally insoluble in dilute acid, alkali and neutral salt solutions, comprising the steps of: firstly, preheating the collagen to 63° C. for 20 minutes in water until shrinkage occurs; secondly, cooling the product of step one to 25° C. and immersing it in acid buffer solution of pH 8; thirdly, treating the product of step two with a proteolytic enzyme selected from a class consisting of trypsin, chymotrypsin, and pancreatin for 48 hours; fourthly, dispersing the product of step three into 0.5% acetic acid solution for six hours for extraction, and fifthly, treating the collioidally dispersed solution thus obtained with pepsin at 25° C. for 24 hours.

References Cited in the file of this patent

Gustavson: The Chemistry and Reactivity of Collagen, Academic Press Inc., New York (1956), pp. 260 to 270.